# COMPONENTS:

- (1) Holmium chloride; HoCl<sub>3</sub>; [10138-62-2]
- (2) Ethanol; C<sub>2</sub>H<sub>6</sub>O; [64-17-5]
- (3) Water; H<sub>2</sub>0; [7732-18-5]

# ORIGINAL MEASUREMENTS:

Sakharova, Yu.G; Ezhova, T.A.

Zh. Neorg. Khim. <u>1976</u>, 21, 551-4; Russ. J. Inorg. Chem. (Engl. Transl.) <u>1976</u>, 21, 296-8.

#### VARIABLES:

Temperature

#### PREPARED BY:

T. Mioduski and M. Salomon

# EXPERIMENTAL VALUES:

solubility of HoCl<sub>3</sub>.6H<sub>2</sub>O in 96.8 % C<sub>2</sub>H<sub>5</sub>OH<sup>a</sup> sample 2 sample 1 sample 3 sample 4 mean solubilities mol kg<sup>-1c</sup> g/100 g<sup>b</sup> t/°C g/100 g g/100 g g/100 g g/100 g 20 34.62 34.81 34.86 34.87 34.79 1.406 33.72 33.85 34.04 33.89 33.87 1.350 30 40 33.88 34.02 33.83 34.16 33.97 1.356 34.56 1.388 34.25 34.68 34.47 34.49 50 35.77 35.83 1.478 60 36.02 36.11 35.93

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Isothermal method used. Equilibrium was reached after 3-4 h. Identical results obtained by approaching equilibrium from above and below. Two of the data points in the table obtained after 3 hours of equilibration, and the remaining two data points obtained after 4 h of equilibration.

The metal content in each aliquot taken for analysis was determined by complexometric titration with Trilon B.

Analyses of the solids withdrawn at 20°C, 40°C and 60°C showed the solid phase to be the hexahydrate: i.e. ethanol was not found in any of the solid phases.

SOURCE AND PURITY OF MATERIALS: HoCl<sub>3</sub>.6H<sub>2</sub>O prepd by dissolving c.p. grade oxide in dil (1:3) HCl followed by evapn and crystn. The crystals were dried in a desiccator over CaCl2,  $P_2O_5$  and NaOH. The crystals analyzed for the metal by titrn with Trilon B, and for Cl by the Volhard method. The hexahydrate melted at 163.4 - 164.5°C. 96.8% ethanol prepd by prolonged boiling of c.p. grade 93.5% ethanol with anhydr CuSO4 followed by distn. Ethanol concn detd refractometrically and pycnometrically.

# ESTIMATED ERROR:

Soly: results apparently precise to within  $\pm$  0.9% (compilers).

Temp: nothing specified.

 $<sup>^{</sup>m a}$ It is not clearly stated whether the mixture is 96.8 mass % or 96.8 volume % ethanol.

 $<sup>^{</sup>m b}$ Solubilities reported as grams of hexahydrate in 100 g of solvent.

<sup>&</sup>lt;sup>C</sup>Molalities calculated by the compilers.

COMPONENTS:			ORIGINAL MEA	SUREMENTS:	7
(1) Holmium chlori	de; HoCl.;		Kirmse, E.		
[10138-62-2]	,		Tr. II Vse	s. Konk. po	Teor. Rastvorov
(2) Alkoxy-ethanol	.s		<u>1971</u> , 200-		
(-, ,					
VARIABLES:			PREPARED BY	· · · · · · · · · · · · · · · · · · ·	
T/K = 298			T. Miodusk	i and M. Sal	omon
EXPERIMENTAL VALUES:					
				lubility <sup>a</sup>	
solvent			mass %	mol kg <sup>-1</sup>	nature of the solid phase
2-methoxyethano1;	c <sub>3</sub> H <sub>8</sub> O <sub>2</sub> ;	[109-86-4]	3.2	0.122	$HoC1_3.nC_3H_8O_2$ (n = 2-3)
2-ethoxyethanol;	C4H <sub>10</sub> O <sub>2</sub> ;	[110-80-5]	8.4	0.338	HoC1 <sub>3</sub> .2C <sub>4</sub> H <sub>10</sub> O <sub>2</sub>
		AUXILIARY	INFORMATION		
METHOD/APPARATUS/PRO		hut rose		PURITY OF MA	•
Experimental detail probably similar to author which are co volume.	previous wor	ks of the	previous salt was		author, the anhydrous epared by the method
			ECTIMATED P	PPOP.	
			ESTIMATED E		
				RROR: specified.	
			Nothing	specified.	
			Nothing REFERENCES: 1. Taylor,	specified.  M.D.; Carte	r, C.P. m. <u>1962</u> , 24, 387.

# COMPONENTS: (1) Holmium chloride; HoCl<sub>3</sub>; [10138-62-2] (2) 1,2-Diethoxyethane; C<sub>6</sub>H<sub>14</sub>O<sub>2</sub>; [629-1]-1] VARIABLES: T/K = 298 CRIGINAL MEASUREMENTS: Kirmse, E.M.; Zwietasch, K.J. Z. Chem. 1967, 7, 281. PREPARED BY: T. Mioduski

#### EXPERIMENTAL VALUES:

The solubility of HoCl3 in 1,2-diethoxyethane at 25°C was reported to be

0.37 mass %

The corresponding molality calculated by the compiler is

0.0137 mol kg<sup>-1</sup>

The composition of the solid phase was given in terms of the Ho:C1:ether ratio as 1:2.97:1.82

# AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Isothermal method used. The anhydrous mixtures were equilibrated at 25°C for several days with frequent shaking.

The solid phase was dried in a vacuum desiccator over  $P_2 O_5$ .

Ho was determined by complexometric titration using Xylenol Orange indicator. Chloride was determined by the Volhard titration method.

# SOURCE AND PURITY OF MATERIALS:

Sources and purities of materials not given. The anhydrous chloride was obtained by the method of Taylor and Carter (1).

The solvent was prepared by the Williamson synthesis: i.e. by reaction of  $C_2H_5I$  with the monoethylether of ethylene glycol.

#### ESTIMATED ERROR:

No estimates possible.

#### REFERENCES:

Taylor, M.D.; Carter, C.P.
 J. Inorg. Nucl. Chem. 1962, 24, 387.

EXPERIMENTAL VALUES:

COMPONENTS:  (1) Holmium chloride; HoCl <sub>3</sub> ;  [10138-62-2]  (2) Alkyl ethers	ORIGINAL MEASUREMENTS: Kirmse, E.M.; Dressler, H. Z. Chem. <u>1975</u> , 15, 239-40.
VARIABLES:  Room temperature: T/K = 293-298	PREPARED BY:  M. Salomon and T. Mioduski

ı					
				HoCl <sub>3</sub> solub	ility <sup>a</sup>
	solvent			mass %	mol kg <sup>-1</sup>
	1-methoxypentane; b	с <sub>6</sub> н <sub>14</sub> 0;	[628-80-8]	2.4 <sup>b</sup>	0.091
	1-methoxyheptane;	с <sub>6</sub> н <sub>18</sub> 0;	[629-32-3]	1.0	0.037
	1-methoxyoctane;	с <sub>9</sub> н <sub>20</sub> 0;	[929-56-6]	1.55	0.058
	1-methoxynonane;	c <sub>10</sub> H <sub>22</sub> O;	[7289-51-2]	1.8	0.068
	1-methoxydecane;	с <sub>11</sub> н <sub>24</sub> 0;	[7289-52-3]	2.5	0.095

<sup>&</sup>lt;sup>a</sup>Molalities calculated by the compilers.

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were agitated at room temperature until the solutions were saturated. The anhydrous reagents were handled in a dry box containing  $P_2O_5$ .

Holmium was determined by complexometric titration using Xylenol Orange indicator.

The reported solubilities are mean values based on four determinations for each system.

# SOURCE AND PURITY OF MATERIALS:

No information given.

# ESTIMATED ERROR:

No information given.

#### REFERENCES:

1. Kirmse, E.M. Tr. II. Vses. Konf. po Teor. Rastvorov 1971, 200-6.

<sup>&</sup>lt;sup>b</sup>Identical result for this system reported in (1)

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Holmium chloride; HoCl <sub>3</sub> ; [10138-62-2]	Kirmse, E.M.; Zwietasch, K.J.; Tirschmann, J.; Oelsner, L.; Niedergeases, U. Z. Chem. 1968, 8, 472-3.
(2) Ethers	Kirmse, E.M. Tr. II Vses. Konf. po Teor. Rastvorov. 1971, 200-6.
VARIABLES:	PREPARED BY:
Room temperature: T/K around 298	T. Mioduski and M. Salomon
EXPERIMENTAL VALUES:	

			solubility	a,b
solvent			mass %	mol kg <sup>-1</sup>
1-ethoxy-2-methoxyethane;	c <sub>5</sub> H <sub>12</sub> O <sub>2</sub> ;	[5137-45-1]	0.65	0.024
di-n-propyl ether;	C <sub>6</sub> H <sub>14</sub> O;	[111-43-3]	0.1	0.004
1-ethoxybutane;	C <sub>6</sub> H <sub>14</sub> O;	[628-81-9]	0.01	0.0004
1-methoxypentane;	C <sub>6</sub> H <sub>14</sub> O;	[628-80-8]	2.4	0.091
1,4-dioxane;	C4H802;	[123-91-1]	0.55	0.020

 $<sup>^{</sup>m a}$ Molalities calculated by the compilers.

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

The solute-solvent mixtures were isother-mally agitated at 25°C or at room temperature. Authors state that the difference found for the solubility was within experimental error limits.

Ho was determined by complexometric titration.

No other details given.

#### SOURCE AND PURITY OF MATERIALS:

The anhydrous salt was prepared by the method of Taylor and Carter (1).

No other information given.

# ESTIMATED ERROR:

Nothing specified.

#### REFERENCES:

Taylor, M.D.; Carter, C.P.
 J. Inorg. Nucl. Chem. <u>1962</u>, 24, 387.

<sup>&</sup>lt;sup>b</sup>Nature of the solid phases not specified.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Holmium chloride; HoCl <sub>3</sub> ; [10138-62-2]	Rossmanith, K.; Auer-Welsbach, C. <i>Monatsh. Chem.</i> <u>1965</u> , 96, 602-5.
(2) Tetrahydrofuran; C <sub>4</sub> H <sub>8</sub> O; [109-99-9]	
VARIABLES:	PREPARED BY:
Room Temperature: T/K about 293	T. Mioduski

#### EXPERIMENTAL VALUES:

The solubility of  $HoCl_3$  in tetrahydrofuran at  $20^{\circ}C$  (room temperature) was reported to be

0.698 g per 100 m1 of solution (0.0257 mol  $dm^{-3}$ , compiler).

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Isothermal method employed. The solution was equilibrated in an extractor with agitation for 60-80 hours at room temperature.

Holmium was determined by the oxalate method and by titration with EDTA using Xylenol Orange indicator. The solvent was determined by difference.

Anhydrous materials were handled in a dry box through which was passed a stream of nitrogen free of carbon dioxide.

The solid phase is  $HoCl_3.3.33C_4H_80$ .

# SOURCE AND PURITY OF MATERIALS:

Sources and purities of initial materials not specified. HoCl<sub>3</sub> was prepared by conversion of the oxide by high temperature reaction with an excess of NH<sub>4</sub>Cl followed by heating the product in a stream of dry nitrogen, and then in vacuum to remove unreacted NH<sub>4</sub>Cl.

Tetrahydrofuran was distilled from LiAlH4.

ESTIM	ATED	ERRO	DR:

Nothing specified.

#### COMPONENTS: ORIGINAL MEASUREMENTS: Korovin, S.S.; Galaktionova, O.V.; Holmium chloride; HoCl<sub>3</sub>; Lebedeva, E.N.; Voronskaya, G.N. [10138-62-2] Zh. Neorg. Khim. 1975, 20, 908-14; Russ. Tributylphosphate; C12H27O4P; (2) J. Inorg. Chem. (Engl. Transl.) 1975, 20, [126-73-8] 508-11. VARIABLES: PREPARED BY: T. Mioduski and M. Salomon T/K = 298

#### EXPERIMENTAL VALUES:

#### Composition of saturated solutions

mass %	mol/kg sln	g dm <sup>-3</sup>	mol dm <sup>-3</sup>	mol kg <sup>-1</sup> (compilers)	density/g cm <sup>-3</sup>
41.0	1.52	573.1	2.12	2.56	1.39

The solid phase is HoCl3

#### AUXILIARY INFORMATION

#### METHOD/APPARATUS/PROCEDURE:

Saturated solutions prepared isothermally with magnetic stirring. Equilibrium was attained after 25-30 d. The solution was centrifuged and an aliquot for analysis taken and added to methanol and precipitated with aq NH3. The pptd Ho(OH)3 was washed repeatedly and heated to the oxide for gravimetric analysis. The solid phase was analyzed (no details given) for phosphorous and only the anhydrous HoCl3 was found.

All operations were performed in a dry box through which a stream of argon was passed.

The major objective of this work was to establish the nature of complexation between TBP and HoCl3 in solution.

# SOURCE AND PURITY OF MATERIALS:

Anhydrous HoCl<sub>3</sub> prepared by chlorination of the oxide with CCl<sub>4</sub> vapor (1,2). Source and purity of materials not given. Ho was analyzed gravimetrically, and Cl by Volhard's method.

Tributylphosphate (TBP) was purified "by the standard method." No additional details given.

#### ESTIMATED ERROR:

No estimate possible.

- Korshunov, B.G.; Drobot, D.V.; Bukhtiyarov, V.V.; Shevtsova, Z.N. Zh. Neorg. Khim. 1964, 9, 1427.
- Novikov, G.I.; Tolmacheva, V.D. Zh. Prikl. Khim. 1965, 38, 1160

# 318 Holmium Chloride ORIGINAL MEASUREMENTS: COMPONENTS: (1) Holmium chloride; HoCl3; Kirmse, E.M. [10138-62-2] Tr. II Vses. Konf. po Teor. Rastvorov (2) Alkyl amines 1971, 200-6. VARIABLES: PREPARED BY: T/K = 298T. Mioduski and M. Salomon EXPERIMENTAL VALUES: HoCl<sub>3</sub> solubility<sup>a</sup> mol kg<sup>-1</sup> mass % solvent [107-10-8] 33.4 1.849 1-propanamine; $n-C_3H_9N$ ; 1.2 0.045 (sec-C<sub>3</sub>H<sub>9</sub>N)<sub>2</sub>NH; [626-23-3] di-2-butylamine; <sup>a</sup>Molalities calculated by the compilers.

# AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Experimental details not given, but were probably similar to previous works of the author which are compiled throughout this volume.

Nature of solid phases not specified.

# SOURCE AND PURITY OF MATERIALS:

Nothing specified, but based on previous work by the author, the anhydrous salt was probably prepared by the method of Taylor and Carter (1).

# ESTIMATED ERROR:

Nothing specified.

# REFERENCES:

Taylor, M.D.; Carter, C.P.
 J. Inorg. Nucl. Chem. <u>1962</u>, 24, 387.

#### COMPONENTS:

- (1) Holmium chloride; HoCl<sub>2</sub>; [10138-62-2]
- (2) Hexamethylphosphorotriamide; C<sub>6</sub>H<sub>1,8</sub>N<sub>3</sub>OP; [680-31-9]

# ORIGINAL MEASUREMENTS:

Mikheev, N.B.; Kamenskaya, A.N.; Konovalova, N.A.; Zhilina, T.A.

Zh. Neorg. Khim. <u>1977</u>, 22, 1761-6; Russ. J. Inorg. Chem. (Engl. Transl.) <u>1977</u>, 22, 955-8.

#### VARIABLES:

Room temperature:  $T/K = 298 \pm 3$ 

#### PREPARED BY:

T. Mioduski and M. Salomon

#### EXPERIMENTAL VALUES:

Starting with the solvate  $HoCl_2$ .  $3C((CH_2)_2N)_2PO$ , the solubility at 25  $\pm$  3°C<sup>a</sup> was given as

 $0.108 \text{ mol dm}^{-3}$ 

 $^{a}$ Table 3 in the English translation of the source paper states the temperature to be 23  $\pm$  3°C. This is probably a typographical error as the text clearly states that all measurements were carried out at 25  $\pm$  3°C.

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Isothermal method. Salt and solvent were placed in a test-tube in a dry box, and the tube agitated at room temperature until equilibrium was reached. Aliquots were withdrawn periodically and analyzed for the metal content. Rare earth concentration was determined by complexometric titration, and by the radiometric method using the isotope Tm-170 (t<sub>1</sub> = 169 d). Authors state that results for both methods agreed. Although not clearly stated, it appears that equilibrium was reached in several weeks to several months.

Solid phase samples washed three times with benzene or ether and dried on a steam bath in an argon atmosphere. The solid phase was analyzed and found to be HoCl<sub>3</sub>.3C<sub>6</sub>H<sub>18</sub>N<sub>3</sub>OP.

The solvate was analyzed for metal content by complexometric titration, for chloride by the Volhard method, and the solvent was obtained by difference. IR spectra confirmed the absence of water. Structural studies of the solvate were also carried out by X-ray analysis.

# SOURCE AND PURITY OF MATERIALS:

 ${\rm HoCl}_3.3{\rm C}_6{\rm H}_1{\rm 8}{\rm N}_3{\rm OP}$  prepared by dissolving the hydrate in the solvent and heating to  $140-145^{\circ}{\rm C}$  for 5 m. The solvate was pptd by addition of abs ether, washed 7 times with ether, and dried over  ${\rm P}_2{\rm O}_5$  in a stream of dry nitrogen. Yield was about 90%.

The solvent was purified as described in (1).

#### ESTIMATED ERROR:

Soly: precision  $\pm$  0.001 mol dm<sup>-3</sup> at a 95% level of confidence (authors).

Temp: precision ± 3 K.

# REFERENCES:

 Fomicheva, M.G.; Kessler, Yu.M.; Zabusova, S.E.; Alpatova, N.M. Elektrokhimiya 1975, 11, 163.

# COMPONENTS: (1) Holmium chloride; HoCl<sub>3</sub>; [10138-62-2] (2) Tetrachlorostannate; SnCl<sub>4</sub>; [7646-78-8] (3) Phosphorus oxychloride; POCl<sub>3</sub>; [10025-87-3] VARIABLES: T/K = 293 Concentration of SnCl<sub>4</sub> Concentration of SnCl<sub>4</sub> Concentration of SnCl<sub>4</sub> Concentration of SnCl<sub>4</sub> ORIGINAL MEASUREMENTS: Lyubimov, E.I.; Batyaev, I.M. 7h. Prikl. Khim. 1972, 45, 1176-8. Th. Prikl. Khim. 1972, 45, 1176-8. Th. Prikl. Khim. 1972, 45, 1176-8.

#### **EXPERIMENTAL VALUES:**

SnCl <sub>4</sub> :POCl <sub>3</sub> ratio	SnCl <sub>4</sub> concentration	solubility <sup>a,b</sup>
(by volume)	mol dm <sup>-3</sup>	moles Ho ${\rm dm}^{-3}$
0	0	0.003
1:100	0.085	0.009
1:50	0.17	0.026 (0.015)
1:25	0.33	0.041
1:15	0.59	0.031
1:10	0.78	0.044

<sup>&</sup>lt;sup>a</sup>Solutions preheated to 220°C. Value in parenthesis corresponds to preheating at 120°C.

$$Ho_2O_3 + 6POCl_3 = 2HoCl_3 + 3P_2O_3Cl_4$$

Authors state that the solubility of HoCl3 is enhanced by complex formation according to

$$2HoCl_3 + 3SnCl_4 = Ho_2(SnCl_6)_3$$

#### AUXILIARY INFORMATION

# METHOD/APPARATUS/PROCEDURE:

Isothermal method used. POCl<sub>3</sub> + SnCl<sub>4</sub> solutions were prepared by volume in a dry box. The SnCl<sub>4</sub> content was verified by chemical analysis for Sn. This solution and Ho<sub>2</sub>O<sub>3</sub> were placed in sealed ampoules, heated to 20-250°C to increase the rate of solution, and then rotated in an air thermostat at 20°C for 2-200 hours. Without preheating, equilibrium was established after 200 hours. Prehating to 220°C lowered the equilibration time at 20°C to 2 hours.

Ho was determined by colorimetric analysis or by the oxalate method. The reported solubilities are mean values based on 3-5 parallel determinations.

# SOURCE AND PURITY OF MATERIALS:

 ${
m Ho}_2{
m O}_3$  of "the first sort" was ignited at 950°C for 2 hours.

"Pure" grade  $SnC1_4$  and  $POC1_3$  were dehydrated with  $P_2O_5$  and distilled under vacuum.

#### ESTIMATED ERROR:

Soly: authors state the "coefficient of variance" to be less than 7%.

Temp: precision presumably  $\pm$  0.2K (compiler)

bThis is also the solubility of HoCl3 in the SnCl4-POCl3 mixture because the oxide is quantitatively converted to the chloride according to